Region 10

Lab Notes

Lab NewsLetter

Vol. VI No. 1
January 2001

Information is knowledge. Knowledge is power. Be empowered.

THIS ISSUE

Organic Methods Work Group – What's It About?

By Roy Araki (360) 871-8703

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), more commonly referred to as SW-846, is a collection of EPA approved methods for sampling and analysis under the RCRA program. A few specific methods are required by RCRA; for other EPA functions, SW-846 serves as a guidance document for sampling and analysis. It is a document that is continually changing with revisions to existing methods for improved performance, incorporating of new methods to support changes in the regulatory program and new technologies that benefit the program.

When new methods or changes to methods are proposed for SW-846, they are announced in the Federal Register (FR) and published as "proposed updates" to SW-846. There is a period of time for comment by the public and private sectors. After EPA has addressed all of the comments, the "final update" is announced in the FR and incorporated as a final rule into RCRA.

Before methods reach the "proposed update" stage, they are subjected to a rigorous review process. The task of review falls on the shoulders of the organic and inorganic methods work groups. These groups consist mainly of senior chemists from the regional laboratories. It is their responsibility to review all proposed methods for program relevancy, qualitative and quantitative ruggedness, and just plain old good science. The work groups meet every July in Washington D.C. and have "as needed" sporadic tele-conferences as methods surface.

It can easily take a year or two before methods make their way through a work group. The Manchester Laboratory first brought its GC-AED method 8085 before the organic methods work group in 1994. It was officially put on the agenda for the July 1995 meeting where the work group requested an inter-laboratory study to satisfy the method validation process before further consideration. The study was completed and presented to the work group at the July 1997 meeting. The method was well received and based on the supporting inter-laboratory data, the work group suggested it be upgraded from a screening method to a full quantitative method. This revision necessitated major re-writing with the method finally being recommended for "proposed update" status in update IVB and placed on the SW-846 web site in late 1998.
(See ***Organic Methods****)*

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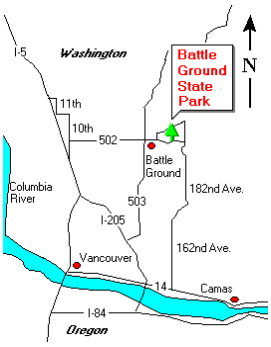
Funky Beaches *E. coli* and Recreation

By Stephanie Harris (360) 871-8710

Battle Ground Lake, formed by the remnant of a volcanic cone, is located southwest of Vancouver, WA in Clark county. This recreational lake, part of a popular state park, is used for fishing and swimming, particularly during the dog-days of summer. In August 1999, Battle Ground Lake was the site of a waterborne disease outbreak that left 32 people sick, seven hospitalized and at least 3 children with kidney failure. The organism responsible for the outbreak was *E. coli* O157:H7, a bacteria best known in the Northwest as the "Jack-in-the-Box" strain after its implication in a serious food-borne outbreak that occurred here in 1993. During this recent outbreak, two independent laboratories isolated this strain from sediments in Battle Ground Lake and from the freshly formed feces of a duck at the lake. Using current molecular techniques, the laboratories were able to determine that the strain found in the sediments and the duck were identical and the cause of the human illnesses. The most likely source of the organism is thought to have been contamination by human feces. As a result of the outbreak, Battle Ground Lake was closed to swimming, boating and fishing during the investigation.



Before this outbreak, the Clark County Health Department monitored the lake water for fecal Coliform bacteria. Not surprisingly, the lake water showed acceptable levels of these bacteria. Fecal Coliform had for years been considered a good indicator of recent fecal contamination by warm-blooded animals. However, studies done by EPA and others later found a poor association between the presence of fecal Coliform bacteria in recreational water and the incidence of illness in swimmers. Other bacteria, such as *enterococci*, demonstrate a better association. In 1986, EPA published a guidance document recommending a switch from fecal Coliform to these other indicator bacteria for monitoring recreational waters to better safeguard public health. *(See* ***Beaches****)*

A Lab in the Field The MuthMobile

By Gerald Dodo (360) 871-8728

In late autumn of 1998, Manchester's field presence was enhanced dramatically with the acquisition of a new mobile laboratory. A truck-mounted laboratory 30 feet in length, the mobile lab has the following major features: diesel powered truck, cables for accessing shore power, on-board power generators, epoxy counter tops with anchoring system for instruments, epoxy floor, fume hood, 25 gallon water supply and waste water storage, water purification system, stainless steel sink, laminate walls and ceiling, hydraulic stabilizing jacks, air-conditioning system, forced air heating system, vehicle and laboratory security alarm system, refrigerator, exterior flood lights, and extensive cabinetry. It was designed to accommodate modern analytical instrumentation such as gas chromatograph (GC) and atomic absorption systems, sample preparation and data processing.

This new mobile laboratory has been dubbed the MuthMobile, after Gerald Muth, the initiator of the acquisition of the mobile lab. The MuthMobile provides sophisticated analytical capabilities for trace level analyses on-site providing analytical data within a few to <24 hours from the time of sample collection. It is currently being used and maintained by the Environmental Services Assistance Team (ESAT) contractor in support of the Region 10 Superfund Program. During fiscal year 1999, ESAT used the MuthMobile to provide more than 1200 sample analyses for either polynuclear aromatic hydrocarbons (PAH), pentachlorophenol (PCP), selected chlorinated volatile *(See* ***MuthMobile****)*

PCB Congener Method Development

By Bob Rieck (360) 871-8719

Polychlorinated biphenyls (PCBs) are thermally stable and have anti-arcing capability. Because of these characteristics, they have been used extensively as dielectric fluids in transformers, capacitors, and fluorescent light ballasts. PCBs have also been used as carrier solvents for ink, pesticides, carbon-less copy paper and as plasticizers to name some other uses. PCBs are extremely persistent in the environment, like the chlorinated pesticide DDT. Like DDT, PCBs also accumulate in fatty tissues. In fact, all of the 300 or so fish samples collected in a recent Region 10 study contained detectable PCB residues, although most samples were quite low. This was also true in a study conducted a couple years ago at Cook Inlet, Alaska by EPA's Office of Water.

PCBs were manufactured until October 1977, mainly by Monsanto, which marketed them under the registered trademark Aroclor®. They were formed by reacting chlorine with a polyaromatic hydrocarbon in the presence of an iron catalyst and heat. The product was distilled into discreet distillation ranges defined for a particular Aroclor. Aroclor® 1260, for instance, had a distillation range of 385-420°C, and Aroclor® 1242 of 325-366°C. These differences in distillation range were important in selecting an Aroclor® for a particular application. The first two digits of the Aroclor® designation refer to the base structure- in the case of PCB's, a biphenyl (12 carbons). The last two digits are *(See* ***PCB****)*

Organic Methods *(continued)*
At the present time Manchester is involved in several other projects that may find their way into SW-846. The lab is providing technical support for the development of an immuno-assay method for the explosives TNT and RDX. This method was "tabled" at the last work group teleconference pending more work on method reliability. Manchester completely rewrote the calibration section of the generalized chromatography method 8000. This revision was submitted just recently for the work group's consideration. The lab is also working on a sample preparation protocol for separation of PCB congeners (see Bob Rieck's article in this issue), a procedure eagerly anticipated by many of the members of the organic methods work group. On a more fundamental level, our regional Quality Assurance Unit recently raised a question about basic chromatographic data acquisition. During the elution of a chromatographic peak are 5 sampling points enough to describe the shape of the peak? This is an important question with significant ramifications for methods using digitized data acquisitions systems. The lab is investigating this issue in hopes of developing guidance that will be included in the many relevant existing methods.

Beaches *(continued)*
The occurrence of an outbreak in recreational water meeting the old fecal Coliform standards is not an isolated event. The Agency's concern over health standards associated with recreational waters prompted EPA to implement the Beach Environmental Assessment, Closure and Health (BEACH) Program, a project to monitor recreational beaches in the United States, announced formally on May 23, 1997 by EPA Administrator Carol Browner. This program is designed to encourage governmental agencies at the federal, state, tribal and local level to strengthen beach water quality standards and testing methods, use water pollution models to predict potential problems and to make information about the risks associated with swimming in contaminated beach water available to the public. As part of the BEACH Program, an Internet-based information system is available to inform the public about beach closings and public health advisories. The Internet address for the BEACH homepage is

http://www.epa.gov/OST/beaches/

Frequent monitoring of recreational waters for the presence of these predictive bacteria during the seasons of recreational use is essential to the success of this program. Unfortunately, due to budgetary cutbacks and other drains on their programs, many states in Region 10 have not been able to implement it. EPA Region 10 may be able to help the states determine the degree of risk to swimmers by conducting bacteria and sanitary surveys at selected high risk beaches using the region's mobile microbiology laboratory. A mobile microbiology laboratory has been developed to provide on-site analytical support to the enforcement program's annual CAFO (concentrated animal feeding operation) inspection effort. This laboratory is currently used for analysis of Coliform bacteria as a measurement of recent fecal contamination. The laboratory is truly mobile and could be used to monitor water quality in recreational areas. In addition, as an extension of the on-going education program at the Regional Laboratory, training in the new microbiology methods could be provided to local government and private laboratories upon request.

MuthMobile *(continued)*
organic compounds (tetrachloroethene, trichloroethene and breakdown products), or total petroleum hydrocarbons (TPH) while at Superfund sites in Oregon and Washington. For a few projects, two GC systems were used at the same time due to the need for PAH, PCP and, for one project, TPH results for each sample on-site. PAH, PCP and TPH are useful parameters in the assessment of sites that have or had wood treatment activities present. Tetrachloroethene and trichloroethene are used to assess pollution caused by dry-cleaning operations or de-greasing cleaning solutions. During this same period ESAT also used the RV-style mobile laboratory Gahler, named after the late former Manchester Laboratory Director Arnold Gahler, to provide 820 sample analyses for the herbicide Dinoseb in support of an emergency removal project in south central Washington.

The methods used for these analyses were developed for screening purposes. Confirmation analyses results from other laboratories and through the use of various quality control type samples have indicated that the field generated data were generally accurate and met the projects' needs. Some of the methods were developed or refined to accommodate the specific needs of the projects just prior to mobilization to the site.

Besides the analyses mentioned above, the MuthMobile along with the available instrumentation and ESAT staff have the ability to provide field screening data for metals, chlorinated pesticides, polychlorinated biphenyls (PCBs), benzene, toluene, ethylbenzene, and xylene (BTEX), ethylene dibromide (EDB), and 1,2-dibromo-3-chloropropane (DBCP). Methods for other parameters can also be developed.

With the MuthMobile, we can deliver more analytical capability and capacity on site, processing more samples faster. Along with the Gahler and the truck-mounted Geoprobe direct push-rod system for subsurface soil, sediment, water, and soil gas sampling, the Region has an outstanding array of field services at its disposal. Any questions on ESAT field support should be directed to Gerald Dodo, (360) 871-8728.

Featured Lab Person

Roy Araki has acquired almost thirty years experience in analytical chemistry in many of the areas in our laboratory, including GC/MS, GC, metals, and conventionals analysis. This has been of enormous benefit in his current tour of duty, QA Coordinator for the laboratory. All in all, though, faced with yet another laboratory SOP, we're sure he'd rather be holding his new granddaughter, Valerie.

PCB *(continued)*
the average percent chlorination in the formulation. Thus, Aroclor® 1260 an average of 60% chlorination by weight, Aroclor® 1242 an average of 42%, and both are on a biphenyl base. There are 209 possible individual chlorinated PCBs, different proportions of which comprise each of the commercial Aroclors®. The individual compounds are known as PCB congeners.

The Region 10 Laboratory has analyzed for Aroclors® using conventional gas chromatographic analysis with electron capture detectors since 1971 in support of TSCA and other programs. Aroclor® analysis involves pattern matching of the multiple peaks in a sample compared to the pattern of multiple peaks in a known standard. Pattern matching permits the chemist to both identify the Arochlor® and quantify the amount present. In contrast, PCB congener analysis compares a single, discrete congener peak in a sample to that of the same single peak in the standard. This is analogous to analyzing single component pesticides which also have only one peak for each analyte.

Aroclor® analysis alone is generally no longer considered sufficient information. EPA now requires data for individual PCB congeners, particularly those on the World Health Organization (WHO) list. The more toxic of these congeners, some of which approach the toxicity of 2,3,7,8-tetrachlorodibenzodioxin, perhaps the most toxic chemical man has made, tend to be the "coplanar" PCBs. This refers to the absence of chlorine in the ortho positions of the biphenyl carbon-carbon bond which allows the two aromatic rings of the biphenyl to inhabit the same plane, similar to 2,3,7,8-tetrachlorodibenzodioxin. These congeners are present in very low concentrations in Aroclor® mixes and are masked by higher concentrations of other, less toxic congeners.

Looking for specific congeners is something like "looking for the needle in the haystack." There is only one "approved" method for PCB congener analysis, Method 1668. This method uses a very expensive ($600,000 to $1,000,000) and sensitive high resolution mass spectrometer, requires specially designed clean rooms, and expensive radio-labeled standards.

The Laboratory has developed a separation scheme that shows promise in separating the small amounts of the more toxic PCB congeners from the remainder of the dominant, less toxic PCB congeners. At the request of the EPA Region 2 Laboratory, we analyzed six flounder samples in duplicate for both Aroclors® and PCB congeners last year. One pair of duplicate samples contained high Aroclor® residues and detectable PCB congener #77, one of the coplanar congeners with toxicity known to approach 2,3,7,8-tetrachlorodibenzodioxin. There was excellent agreement between the duplicate values. Moreover, the PCB congener #77 concentration was 0.9% of the Aroclor® concentrations in the duplicates, demonstrating significant amounts of toxic coplanar PCB congeners are present in environmental samples, such as these fish tissues, when appreciable Aroclor® is present

Since we have acquired a newer, more sensitive instrument and most of the 209 congeners, our next step is to evaluate the performance of this new procedure on environmental samples such as sediments and fish tissues and determine method detection limits. The procedure has worked well on NOAA sediment samples and also on the EPA Region 2 fish samples. Our goal is to have the equivalent of a poor person's high resolution mass spectrometer, satisfy the Region's needs in a cost effective manner and make chemists throughout the Agency say, "Why didn't I think of that?"